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13. SUPPLEMENTARY NOTES

14. ABSTRACT

The goal of the project is to examine and understand the effect of NbO additions in refining the grain size of steels and the effect of rare earth additions in alloy steels. The specific objectives are as follows:

- Build an understanding of the underlying mechanism of NbO based grain refinement.
- · Document structure property relations in several material conditions for NbO refined steels.
- Expand the current understanding of rare earth based grain refinement by examining their influence on alloy steels.
- Document structure property relations in several material conditions for rare earth refined alloy steels.

15. SUBJECT TERMS

Steel, grain refinement, solidification, rare earth

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Office of Naval Research (ONR)

Final Report for Grain Refinement of Steels Through Solidification Modification

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What were the major goals and objectives of the project?

The goal of the project is to examine and understand the effect of NbO additions in refining the grain size of steels and the effect of rare earth additions in alloy steels. The specific objectives are as follows:

- Build an understanding of the underlying mechanism of NbO based grain refinement.
- Document structure property relations in several material conditions for NbO refined steels.
- Expand the current understanding of rare earth based grain refinement by examining their influence on alloy steels.
- Document structure property relations in several material conditions for rare earth refined alloy steels.

What was accomplished towards achieving these goals?

All tasks within the original proposal were accomplished. The Foundry Research Group has published several papers related to the research with some awaiting publication.

Task 1 was to examine the relationship between mechanical properties and NbO additions. This was done through a series of plate casting experiments. Heats of 1030 were melted in an induction furnace. Once the heats reached 1720°C, FeSi, FeMn, and graphite were added. While tapping, aluminum shot additions occurred to fully deoxidize the melt. Once the melt in the ladle cooled to 1620°C, the castings were poured. Additions of NbO, NbO₂, or FeNb occurred in the pouring stream. Two plate castings per heat were poured. For oxide additions, 5g and 10g additions of 99 wt. % pure, -325 mesh powder were done. A slightly higher addition rate of 7g and 14g was done in the FeNb heat to ensure the same amount of niobium was added to all heats. A no addition heat was also poured as a control. After cooling for an hour, the castings were shaken out and allowed to cool to room temperature. Tensile bar, metallurgical, and spectrometer samples were then extracted from the plates. A lathe then machined the tensile bar samples into 0.5 inch diameter tensile bars that met ASTM E8. Tensile testing occurred on a 450 kN hydraulic mechanical test frame. The tests followed ASTM E8 guidelines and used gage marks for elongation measurement. An optical emission spectrometer (OES) analyzed the composition of each steel

heat (See Table 1). Optical microscopy characterized the microstructure and grain size measurements using the linear intercept method were done. A SEM with EDS examined the inclusions to determine differences between the different addition types.

Table 1 Composition of each heat.

Heat	C (wt. %)	Si (wt. %)	Mn (wt. %)	P (wt. %)	S (wt. %)	Al (wt. %)	Nb (wt. %)
Baseline	0.28	0.23	0.83	0.006	0.003	0.14	0.006
NbO	0.31	0.22	0.83	0.003	0.004	0.13	0.036
NbO ₂	0.28	0.22	0.79	0.014	0.007	0.14	0.037
FeNb	0.31	0.20	0.76	0.014	0.010	0.13	0.059

The average YS and UTS are plotted in Figure 1 with 95 % confidence intervals for each of the steels poured. Statistically, the UTS of all the steels was the same. However, the niobium containing heats had statistically higher YS. NbO has a crystallographic structure similar to δ -ferrite and was thought to act as a heterogeneous nuclei. NbO₂ and FeNb do not have structures that allow them act as nuclei. Their use in these experiments was to determine if the main increase in strength was due to the presence of niobium in the steel. It was originally theorized that the NbO additions would produce the highest strength. However, the YS are statistically similar and the data hints that NbO₂ and FeNb may have actually produced higher strengths.

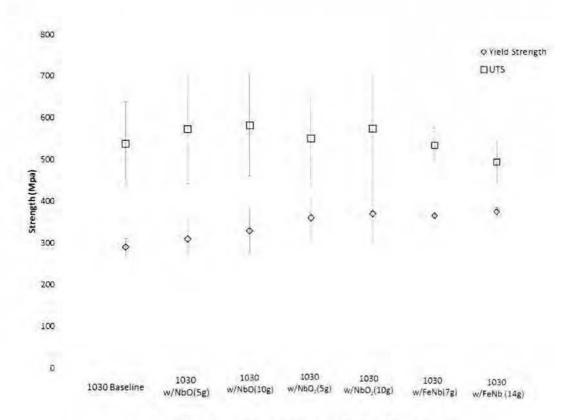
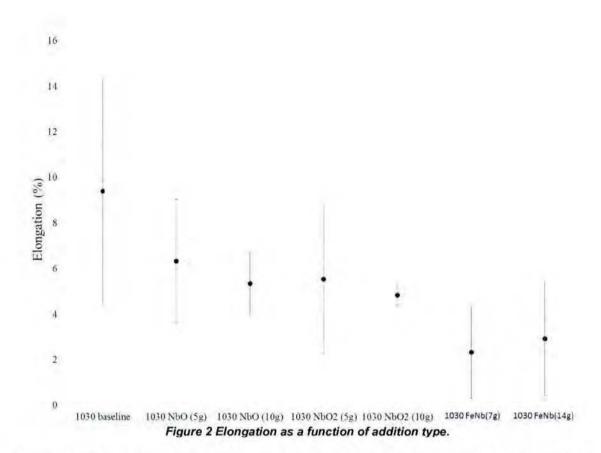


Figure 1 Strength as a function of Nb addition type.



The elongation of the niobium containing heats were lower than the baseline heat (See Figure 2). This does not correspond to the expected behavior if grain refinement through improved nucleation were the driving force behind the strengthening of the steels. Typically, elongation and strength increase together in a Hall-Petch mechanism. Thus, the strengthening observed must be due to another mechanism.

Due to limited thermodynamic data on the stability of NbO and NbO₂ in liquid steel, Task 2 was a set of experiments to determine the thermodynamic stability of these phases. To conduct the experiments, 2.5 g of 99 wt. %, -325 mesh powders of either NbO or NbO₂ were added to 40 g of 99 wt. % pure iron granules in a small alumina crucible. A control experiment was also done with a 3 g mass of industrial FeNb as a control. An alumina cover then was placed on the crucible and this assembly was buried in graphite contained in a larger alumina crucible (See Figure 3). This was inserted into a resistance furnace and heated at a rate of 5°C/min until 1590°C. The samples remained at 1590°C for two hours and then cooled to room temperature over eight hours. After a run, the samples were sectioned into metallographic specimens and analyzed in the optical and scanning electron microscopes. In none of the equilibrium experiments did the original niobium containing phase remain. The evidence indicates that all of the compounds dissolved in the steel melt. Oxygen from the NbO or NbO₂ powders reacted with other elements to form complex oxides with far lower niobium content than the original powders. These experiments indicate that none of the niobium containing compounds were thermodynamically stable in molten steel.

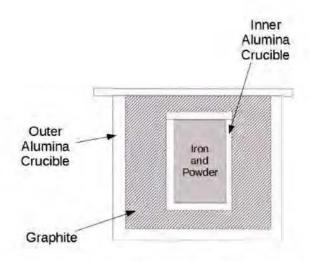


Figure 3 Schematic of the phase equilibrium setup.

Task 3 centered on the creation and use of a method to remove oxide inclusions from solid steel to provide a better path for characterizing them. Two methods were examined during a literature review, acid or bromine dissolution of the steel and electrolytic dissolution. Acid or bromine dissolution methods were ruled out since they have unstable dissolution rates, frequently dissolve certain classes of inclusions, and present safety hazards. Electrolytic dissolution was selected as the best method to attempt. The electrolytic dissolution apparatus consisted of a power supply, stand, beaker, and alligator clips. A 3.15 mm diameter, 450 mm long graphite rod was attached to the cathode (-) side of the circuit. Then, a 15 mm square, 10 mm thick piece of test steel was placed to the anode (+) part of the circuit. Once both electrodes were placed into a beaker with the electrolyte, a voltage of 3-3.5 V and 40-45 mA current was maintained during an experimental run. Initially, two different electrolytes were evaluated: 10% AA (10 v/v% acetylacetone-1v/v% tetra methylammonium chloride-methanol) and 2% TEA (2 v/v% triehtanolamine-1 w/v% tetramethylammonium-methanol). A sample of steel was weighted at 0, 2, 4, 6, 8, 10, 12, 14, and 16 hours to determine the dissolution rate. At each weighing, the sample was cleaned with acetone and dried before continuing.

Figure 4 depicts the dissolution rate for both solutions. Initially, the team focused on controlling the voltage and had a wider tolerance on current. It was realized when plotting the data in Figure 9 that the current plays a critical role in controlling the dissolution rate. A shift to primarily controlling the current resulted in a much more consistent dissolution rate. The dissolution rate for the AA and TEA were 0.00095 g/min and 0.00071 g/min. Janis et al. had experimented with similar steel samples and electrolytes. In their work, the dissolution rate for 10% AA varied from 0.0004-0.001 g/min. They recommended 2% TEA as a electrolyte because it had a lower variation in dissolution rate. There was no reporting on the current settings used and it is likely that they did not control the dissolution rate using current, but by voltage. The PI's work shows that both electrolytes were very repeatable. The PI chose to move forward with 2% TEA since it's slower dissolution rate allows better control of the volume of material dissolved.

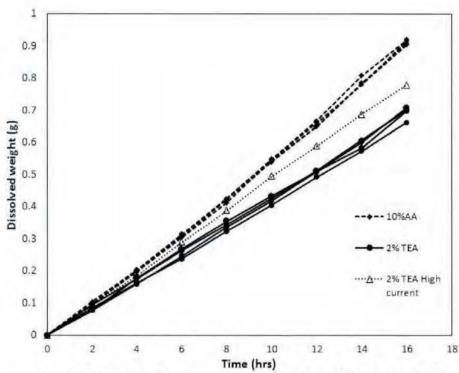


Figure 4 Electrolytic dissolution rate of 1030 steel in 10% AA and 2% TEA.

Samples from the heats poured in Task 1 were processed using electrolytic dissolution and then filtered on a polycarbonate filter with a 0.05 μ m pore diameter. The film was then cut so it could fit in the SEM and carbon coated. SEM results from analysis on the filtered inclusions and those from polished metallurgical samples were compared. The inclusions were larger on the filtered samples when compared to the as-polished specimens (See Figure 5). This has been a common observation in inclusion separation techniques since the actual size of the inclusion can be masked from observation by the matrix. EDS analysis of the polished sections found that the composition of the inclusions was high in niobium and the remaining elements were iron, aluminum, manganese, and oxygen. The compositions were somewhat similar for the filtered samples, but silicon was also detected. Like the inclusion analysis done in Tasks 1 and 2, this technique found very similar oxides formed in the NbO, NbO₂, and FeNB addition steels. None of these oxides had a composition close to that of NbO or even NbO₂. These results reinforce the observation that NbO and NbO₂ are not stable in liquid steel and are unlikely to form. Thus, grain refinement via nucleation of δ -ferrite on NbO is unlikely and the observed strength increase was likely due to the formation of niobium precipitates.

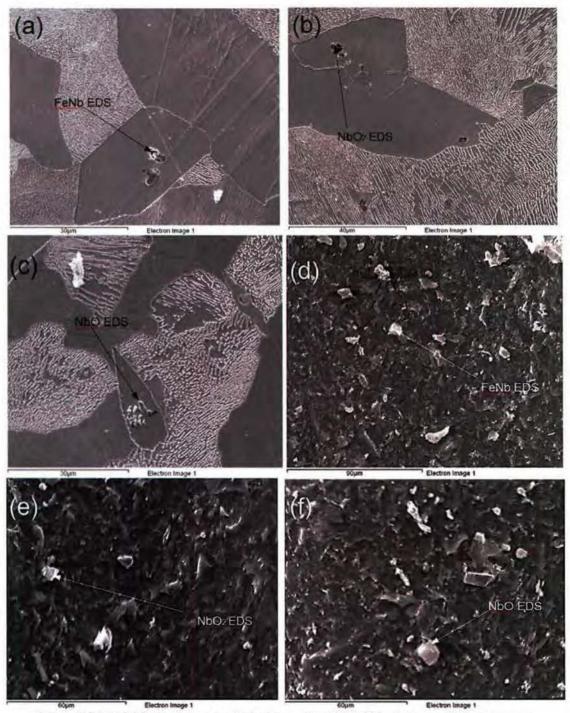


Figure 5 (a) (b) (c) Inclusions on polished samples (d) (e) (f) Inclusions of filtered paper.

Task 4 consisted of examining rare earth additions to 4130 steel by pouring a series of plate casting experiments. These castings were created by melting heats of 4130 in a 3 kHz induction furnace and then pouring into green sand molds. Figure 6 depicts the casting geometry used in those experiments. Additionally, several other pieces of work have been completed or will be finished shortly. Experiments on 1030, 1040, and 1050 were done to determine the effect of austenite fraction on grain refinement response. While conducting the work on quench and tempered 4130, the PI noticed high levels of

microporosity on the fracture surfaces. This lead to a reexamination of the casting geometry and work on developing a keel block casting to reduce microporosity.

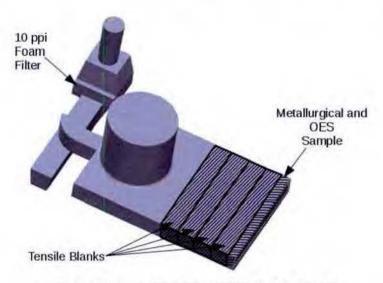


Figure 6 Plate casting geometry used in this project.

Figures 7 and 8 depict the strength and elongation data from the as-cast 4130. Refinement additions of 0.1 and 0.3 wt. % RE silicide or EGR were done for the as-cast case. Steels where RE silicide or EGR had been added had a higher yield strength (See Figure 7). Only for the 0.3 EGR sample was this not statistically significant. The UTS however was lower for all of the addition steels, which may be related to the lower elongations also observed (See Figure 8). The exact cause of the low elongations was not entirely obvious. Examining the samples in the quench and tempered (Q&T) condition found a very different trend. The yield strength, UTS, and elongation of the treated samples were higher than the baseline 4130 (See Figures 9 and 10). Due to significant variation in the baseline data, these trends were statistically insignificant. However, it does fit with previous observations.

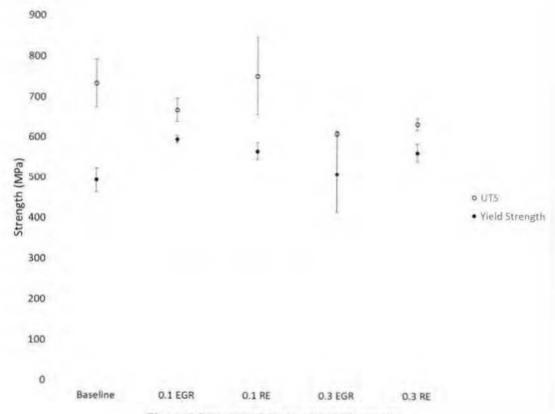


Figure 7 Strength of the as-cast 4130 samples.

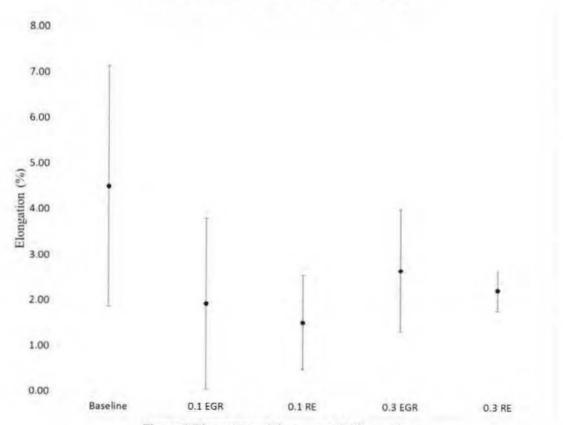


Figure 8 Elongation of the as-cast 4130 samples.

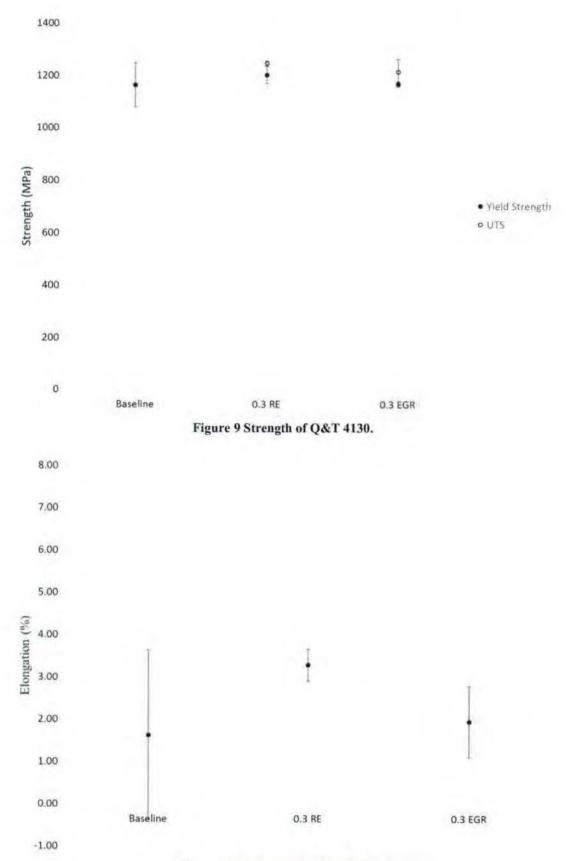


Figure 10 Elongation of Q&T 4130.

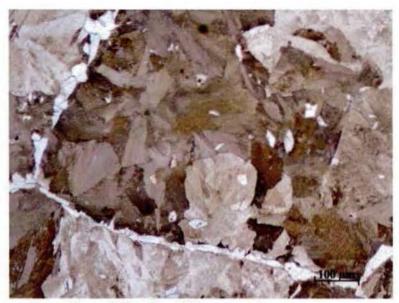


Figure 11 Baseline 4130 as-cast microstructure.

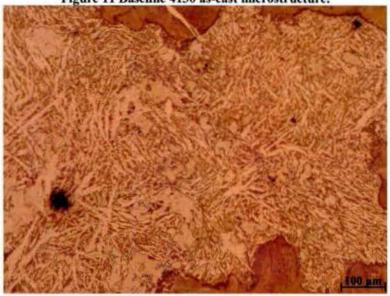


Figure 12 As-cast microstructure of 0.3 EGR 4130.

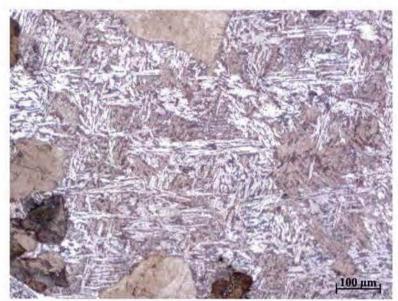
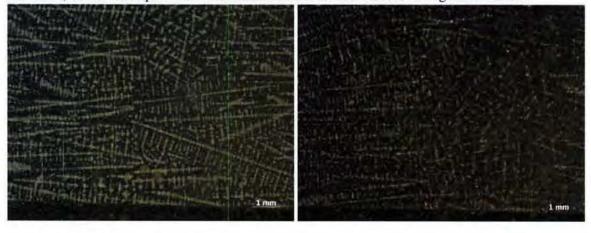


Figure 13 Microstructure of the 0.3 RE 4130 samples in the as-cast state.

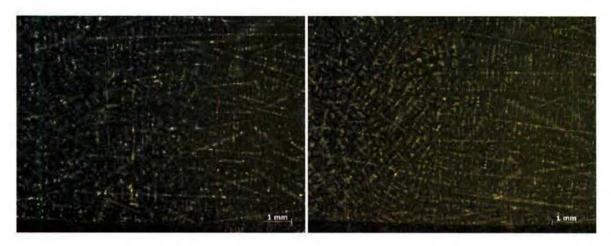
Figures 11 through 13 depict the as-cast microstructures of the baseline, 0.3 wt. % EGR, and 0.3 wt. % RE silicide samples. In the treated samples, a significant change in the microstructure occurred. There were large regions where a fine Widmanstatten ferrite structure occurred (See Figures 7 and 8). Between the Widmanstatten ferrite was very small areas of pearlite. These finer regions were surrounded by more typical pearlite colonies. These fine structures would account for the increase strength, but the cause of the low ductility in the as-cast specimens is not entirely understood by the PI.

Heats of 1030, 1040, and 1050 were poured to examine how the change in primary austenite phase fraction affected a steels response to RE based grain refinement. A baseline and 0. 3 RE silicide version of each alloy were poured. These experiments utilized the old plate casting geometry. Macroetching with Oberhofer's etch revealed that adding 0.3 wt. % RE scilicide refined the solidification structure in all the steels (See Figures 14 through 16). The degree of refinement appeared to be higher as the carbon level increased, which corresponds to an increase in the austenite fraction during solidification.



Baseline 0.3 RE

Figure 14 Macrostructure of the 1030 steels.



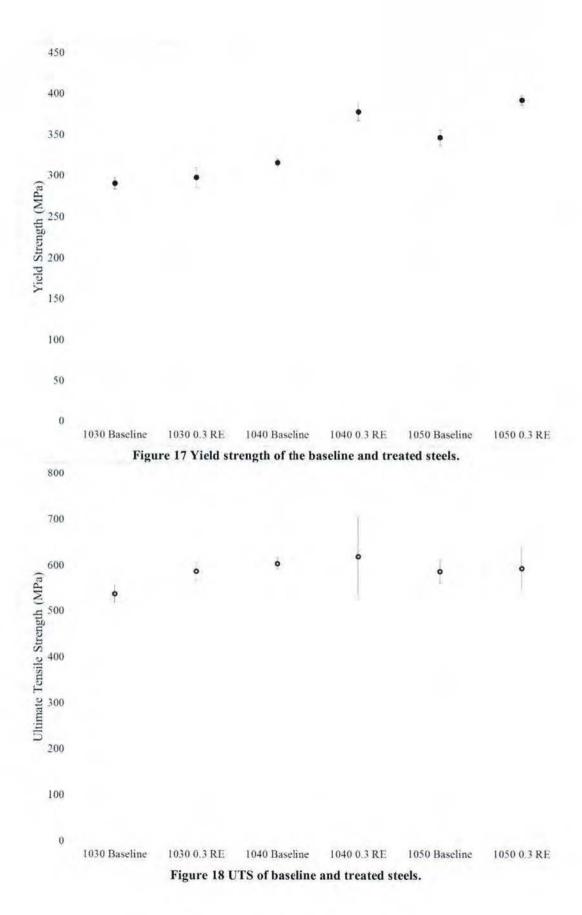
Baseline 0.3 RE

Figure 15 Macrostructure of the 1040 steels.



Baseline 0.3 RE

Figure 16 Macrostructure of the 1050 steels.



Mechanical testing found that strength increased when 0.3 wt. % RE silicide was added to each steel (See Figures 17 and 18). Yield strength in particular was impacted by the RE silicide additions. It was also found that the increase in yield strength became larger as the austenite fraction during solidification also increased. This corresponds with the macroetching results that showed a similar trend. SEM analysis found RE oxysulfides, similar to those already reported, in all of the treated steels. It appears from these experiments that refinement effectiveness improves as the amount of austenite in the steel during solidification increases. This makes sense since many of the RE oxide and sulfide compounds have a better crystallographic match with austenite than δ -ferrite.

For Task 5, a series of HY100 heats were produced using a 3 kHz induction furnace. Green sand molds of the plate casting geometry were made using a jolt/squeeze machine. After the initial melt down of the 1010 charge, ferrochrome, and nickel, the heat was tapped at 1720°C. The final silicon, manganese, and graphite additions occurred just prior to tapping, while the aluminum shot and either 0.3 wt.% rare earth (RE) silicide or EGR were added in the ladle. In addition to the plate castings, a separately cast sample with an elevated phosphorous level was poured to assess the effectiveness of the additions in modifying the solidification structure. Pouring into the molds occurred at 1600°C. The castings cooled for one hour prior to being shaken out. The castings were then sectioned into tensile bar, metallurgical, and optical emission spectrometer samples. Half-inch diameter tensile bars were turned on a lathe in accordance with ASTM E8 and tested on a hydraulic universal testing frame. The metallographic sample and the phosphorous containing bar were further processes into samples and polished. Nital etching of the metallurgical sample provided the as-cast room temperature structure, while etching the phosphorous bars with Oberhofer's Etch revealed the solidification structure.

Figures 19 through 21 present the solidification structure of the samples poured. Neither the 0.3 RE or 0.3 EGR samples had a finer solidification structure than the baseline sample. This indicates that the additions had no effect on the solidification structure and that the RE inclusions found in the samples were not effective nuclei. A Scheil solidification calculation for HY100 indicates that for practically all of its solidification range the solid phase is δ -ferrite. It appears the RE inclusions formed are not effective heterogeneous nuclei for δ -ferrite.

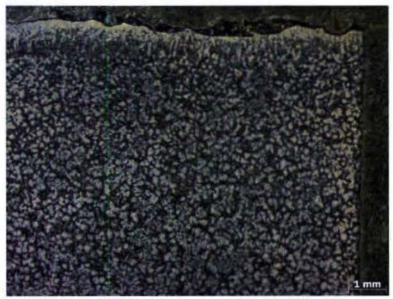


Figure 19 Solidification structure of the HY100 baseline.



Figure 20 Solidification structure of the HY100 0.3 RE sample.



Figure 21 HY100 0.3 EGR sample solidification structure.

Examination of the tensile properties found little difference in the as-cast properties between the steels (See Figure 22). One surprising result was that the elongation of the as-cast material was very low (See Figure 23). However, these were also statistically similar. The mechanical property similarities made sense based on the microstructure similarities. However, the extremely low elongations concerned the PI. Since HY100 is typically used in the quench and tempered (Q&T) condition, samples of each steel were austenized at 900°C for one hour, water quenched, and tempered at 625°C for two hours, as per common industrial practice. Figures 24 and 25 show the mechanical testing results. The treated steels had higher yield strengths and ultimate tensile strengths in the Q&T condition. It may be that the additions do not assist in modifying the solidification structure, but assist in creating a finer austenite grain size upon heat treatment. This finer size then would lead to a finer martensite packet size which may explain the almost 20% increase in strength.

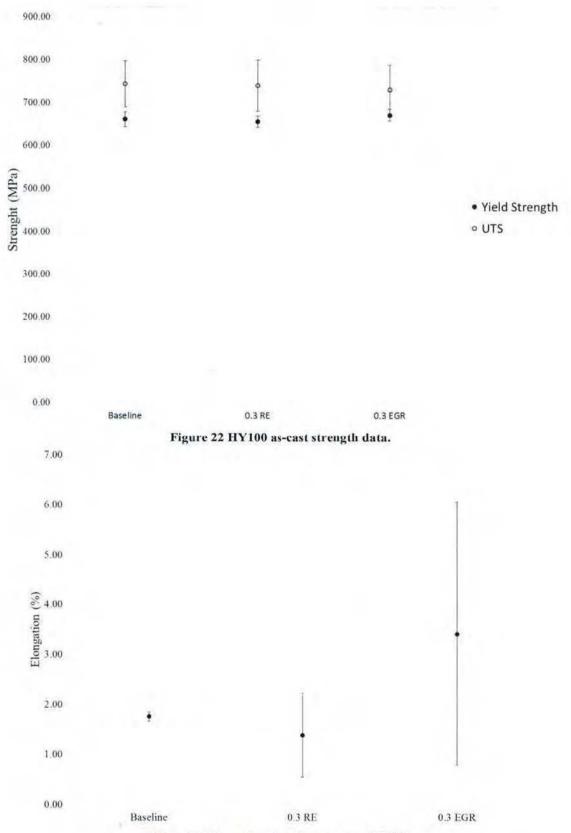


Figure 23 Elongation data from as-cast HY100.

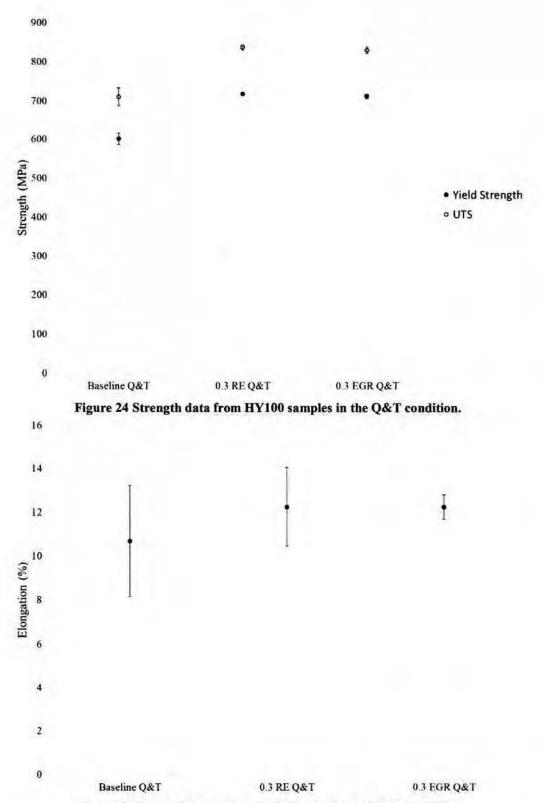


Figure 25 Elongation data from HY100 samples in Q&T condition.

As depicted in Figure 25, the elongations dramatically improved upon quench and tempering. This improvement made little metallurgical sense initially. A tempered martensitic structure should have a

lower elongation than the as-cast structure. This appears to indicate that the as-cast steels contained significant amounts of hydrogen, which was released during the Q&T heat treatment. Future work on another project will confirm this and determine how to remove the hydrogen.

As stated earlier all of the tasks in the project have been completed.

What opportunities for training and professional development did the project provide?

Being associated with a university, one of the primary goals of the Foundry Research Group is to build the skills of its members. This is done in several ways. Each member experiences formal safety training, exposure to new analytical techniques, mentoring, design skill development, learning manufacturing processes, and attendance at conferences. Thus, members learn a large cross section of skills necessary for being an engineer.

Safety has always been the number one priority of the SVSU Foundry Research Group. Each member goes through a rigorous safety training procedure upon entering the group. The first portion of the training focuses on understanding the safety hazards related to molten metal explosions and proper induction furnace practice. This information is presented in a series of online safety videos they must watch. Additionally, training on heat exhaustion is also done. These are followed by an aluminum pour and a cast iron pour. The pours are designed to ensure that all personnel are familiar with pouring procedures and get a chance to work together since the cohesiveness of a pouring crew is vital. Aluminum is done first followed by cast iron. These are done since they are progressively hotter metals. Safety training continues during their entire time; however, this initial training ensures they begin thinking about safety while working and helping students understand that they will be responsible for the safety of others when they work. The training offered follows similar procedures at local companies so it acclimates them to industry standards.

While the graduate and undergraduate programs at SVSU attempt to provide a large exposure to various experimental, manufacturing, and theoretical areas, coverage of every topic is never possible. Students working in the research group learn several topics or gain practical experience in implementing topics covered in their programs. Microscopy receives minor coverage in graduate and undergraduate coursework. Undergraduates learn in their Principles of Engineering course how to etch a sample and capture an image, but quantitative microscopy is not covered. Graduate students learn about some quantitative microscopy techniques, but only in a few homework problems. Both sets of students assist in polishing, etching, and capturing microstructure images. This exposure assists them in developing a broader background in metallography. They also learn how to conduct quantitative microscopy measurements by conducting grain size measurements. Changes in areas being sampled and the interpretation of the microstructure are also covered. Another area where significant materials education occurs is the area of sand testing. Every member of the group learns how to run compactibility, green compressive strength, moisture, and methylene blue tests. These are used in controlling the green sand system to assure molds made in the foundry are the same each experiment. The students also produce

SPC control charts to look for long term trends related to the sand system. This data and its interpretation are also discussed as problems arise. The sand system control chart work provides a powerful experience for students that cannot be replicated in the classroom.

A large number of manufacturing processes are used as part of this research project. The most significant process is the metalcasting process itself. Students learn moldmaking techniques that cannot be taught in the classroom, such as the jolt/squeeze machine or resin bonded mold making. They also learn why certain parameters are used at SVSU based on previous or current defect issues. Students learn CNC machining by using the CNC lathe to machine tensile bars, attend TRAKMILL training to learn conversational programing, and by using the various CNC mills to create experimental apparatus. Students receive training on how to use drill presses, grinding benches, lathes, mills, and other shop equipment. Some of this training is provided in the undergraduate program, but is very limited while the work in the group varies more. As a regular part of the group's activities, students also build or repair experimental equipment. This exposure to different manufacturing processes provides a strong background for students when they begin working in industry.

The PI attempts, particularly with the undergraduates, to have each student work on some type of design project related to the research project. This ranges from new equipment to fixtures. Through the project a student's design skills are enhanced as they gain practice in concept generation, understanding realistic constraints, and developing solid models. The PI meets weekly or more often during the design process to see how it is progressing and giving feedback. This feedback relates both to the project itself, but also the student's work on it. These frequently show small deficiencies in their understanding of engineering topics which are addressed via this process. Finally, the students build their design by themselves or with their peers. This results in immediate feedback on the feasibility of the design from a manufacturability standpoint. Many issues are headed off during the review process with the PI. However, some are purposely left in by the PI to help the students learn from their own mistakes or are unforeseen issues. Again, the students are developing better design and engineering skills by doing engineering, even on a small scale.

The last way in which the SVSU Foundry Research Group develops its members is through plant tours and conference attendance. While there is an active American Foundry Society Student Chapter that conducts tours, group members often travel with the PI to foundries or suppliers around the state when the opportunity arises. These are important in two ways. First, the members see how their work will be integrated by industry and improves their understanding of industry. Second, they develop a broader understanding of industry and a network of contacts for post-graduation employment. Funding from the project ensures each member can attend Metalcasting Congress. This is the premier metalcasting industry conference for North America. Here the students attend workshops and talks on the latest developments in the metalcasting industry. Their attendance increases their knowledge of metalcasting and particularly the current state of the art, and allows them to make contacts for post-graduation employment.

The SVSU Foundry Research Group engages in a series of activities to improve and advance its members. These efforts are both formal and informal in nature, but help create an atmosphere of both process and individual continuous improvement. Those hiring former members often note they are very

hands-on, aware of current trends in industry, and highly skilled. Many former members become key personnel at their various companies.

How were the results disseminated to communities of interest?

Results from the project are primarily disseminated through journal publications and conference presentations. The PI selects the journals and conferences based on their audience. Since the project is designed to develop technology for the foundry and steel industries, publications and conferences that those industries read or attend are prioritized over those with audiences of a more academic nature. This strategy has been used to ensure the work is seen and adopted by industry.

Most of the PI's journal publication related to this project are either in the International Journal of Metalcasting and Journal of Materials Engineering and Performance. The International Journals of Metalcasting is run by the American Foundry Society and published by Springer. It is also the World Foundry Organization's official journal. Due to the focus of the research being on foundry related topics, this journal has been published in for its focus on foundry industry related topics. Many foundry technical people read this journal, in fact it is frequently the only peer review journal US industrial people read, articles published here are broadly read by the industry. This helps ensure the knowledge gained from this project becomes integrated into industrial production. Also, the top academic scholars in the foundry industry read this journal. The Journal of Materials Engineering and Performance also has a more industrial readership. It is sponsored by ASM and published by Springer. This journal focuses on the relationship between manufacturing process and properties. As such, it has a broader audience with a similar interest in more industrial based research. Publishing here allows the PI to obtain feedback from those outside the foundry industry.

Much like the target journals, the conferences that the PI attends and presents at are based on ensuring research results are disseminated to industry. The two conferences where the PI typically publishes are the American Foundry Society's Metalcasting Congress and AIST's AISTech. Both are industrial conferences with significant academic researcher attendance. Metalcasting Congress is the premier North American conference for the foundry industry and has attendees from across the world. Both industrial and academic groups are represented here. The academic figures are the key global researchers in the foundry industry. Similarly, AISTech is the main conference for the North American steel industry with attendees from all over the globe. This event also draws academic attendees that are the top key researchers in steel. Attending and presenting at these conferences ensures that the project's results are being directly disseminated to industry with the goal of integrating these more rapidly than a journal only publication strategy. Additionally due to the focused nature of the researchers attending, feedback and commentary on the work emanates from the most appropriate group possible.

Elkem has developed a commercial grain refiner for austenitic steels. They have generously donated samples of this material for testing for almost a decade. A copy of all papers related to rare earth grain refinement have been sent. Due to their industrial experience, this has resulted in a dialog on the interpretation of results and comparison of the effects in austenitic steels with the carbon and low alloy steels in this work. Many of the conversations related to actual industrial experience and cannot be fully disclosed, but does help guide the work and confirm observations that have been made. As the

dissemination plan outlines, the PI's main focus has been on ensuring research results are presented to industry to advance manufacturing processes. It also focuses on obtaining feedback from those academic researchers with applicable experience in either foundry processes or steels. This approach ensures that project results are rapidly known to industry and should assist in their adoption. It also guarantees that as the project progresses result interpretation is correct, alternative solutions to experimental issues are found, and current thoughts on steel solidification are integrated into the research.

What do you plan to do during the next reporting period to accomplish the goals and objectives?

Nothing to report. Final report for project.

Honors: What honors or awards were received under this project in this reporting period?

- 2015 AFS Steel Division Best Paper Award
- 2015 AFS Applied Research Award

Technology Transfer

No patents or licenses have been issued as part of this project. However, there has been conversations with Elkem about the results. These have been done due to their donation of grain refiners and their industrial experience. Copies of papers have been sent to build their understanding of steel grain refinement.

Participants

There are no limits on the number of participants you list for this section; however, you must list participants who have worked one person month or more for the project reporting period. Students are not included in this section.

You have the option of selecting "nothing to report" in this section.

Have on hand the following information for each participant to enter into the report:

1.	Type: Most senior project role					
	Faculty					

- 2. Prefix (optional): Dr.
- 3. First Name: Robert
- 4. Last Name: Tuttle
- 5. Middle Name (optional)
- 6. Suffix
- Nearest person month worked (a person month equals approximately 160 hours of effort, regardless of funding source): 21
- 8. National Academy Member? N
- Country if participant is a foreign collaboratorIf not US based, identify the country of this participant on this project.

Students

Number of undergraduate and graduate STEM participants: 9 Number of participants that received a STEM degree: 7

Products

You have the option of selecting "nothing to report" in this section.

There are no limitations to the number of entries you submit and you can also pull information directly using the publication DOI.

Below is the information detailed for each product submission:

- 1. Publications (publication reference information
 - a. Article Title: Experimental Grain Refiners for Carbon Steels
 - b. Journal: International Journal of Metalcasting
 - c. Authors: Robert Tuttle
 - d. Keywords: steel, grain refinement, solidification, rare earth
 - e. Distribution Statement: No restriction
 - f. Publication Status: Published
 - g. Publication Identifier Type: 1939-5981
 - h. Publication Identifier: https://doi.org/10.1007/s40962-015-0007-1
 - Publication Date: 2016
 - i. Volume: 10
 - k. Issue: 1
 - 1. First Page Number: 21
 - m. Publication Location: N/A
 - n. Acknowledgement of Federal Support? Yes

o. Peer Reviewed? Yes

- Article Title: Study of Electrolytic Dissolution in Steels and Rare Earth Oxide Stability
- b. Journal: International Journal of Metalcasting
- c. Authors: Aravinda Bommareddy, Robert Tuttle
- d. Keywords: dissolution rate, rare earth oxide stability, electrolyte solution
- e. Distribution Statement: No restriction
- f. Publication Status: Published
- g. Publication Identifier Type: 1939-5981
- h. Publication Identifier: https://doi.org/10.1007/s40962-016-0023-9
- i. Publication Date: 2016
- j. Volume: 10
- k. Issue: 2
- First Page Number: 201
- m. Publication Location: N/A
- n. Acknowledgement of Federal Support? Yes
- o. Peer Reviewed? Yes
- a. Article Title: Role of Niobium Oxides on the Strength of Plain Carbon Steels
- b. Journal: International Journal of Metalcasting
- c. Authors: Aravinda Bommareddy, Robert Tuttle
- Keywords: electrolytic extraction, precipitation hardening, electron microscope, niobium oxides, carbon steel
- e. Distribution Statement: No restriction
- f. Publication Status: Published
- g. Publication Identifier Type: 1939-5981
- h. Publication Identifier: https://doi.org/10.1007/s40962-016-0028-4
- i. Publication Date: 2016
- j. Volume: 10
- k. Issue: 2
- 1. First Page Number: 208
- m. Publication Location: N/A
- n. Acknowledgement of Federal Support? Yes
- o. Peer Reviewed? Yes

2. Conference Paper

- a. Title: Effect of Rare Earth Oxides on Rolling Performance
- b. Authors: Robert Tuttle, John Lewandowski, Robert Tomazin
- c. Conference Name: AISTech

- d. Conference Date: May 2015
- e. Conference Location: Clevland, OH
- f. Publication Status: Published
- g. Publication Date: May 2015
- h. Publication Identifier Type: N/A
- Publication Identifier: N/A
- j. Acknowledgement of Federal Support? Yes
- a. Title: Rare Earth Grain Refiner Addition Methods
- b. Authors: Robert Tuttle
- c. Conference Name: 119th Metalcasting Congress
- d. Conference Date: April 2015
- e. Conference Location: Columbus, OH
- f. Publication Status: Published
- g. Publication Date: April 2015
- h. Publication Identifier Type: N/A
- i. Publication Identifier: N/A
- j. Acknowledgement of Federal Support? Yes
- a. Title: Interaction of Rare Earth Oxide with Alumina Refractories
- b. Authors: Aravinda Bommreddy, Robert Tuttle
- c. Conference Name: 119th Metalcasting Congress
- d. Conference Date: April 2015
- e. Conference Location: Columbus, OH
- f. Publication Status: Published
- g. Publication Date: April 2015
- h. Publication Identifier Type: N/A
- Publication Identifier: N/A
- Acknowledgement of Federal Support? Yes
- a. Title: Strengthening Mechanism in 1030 Steel by Niobium Oxides
- b. Authors: Robert Tuttle
- Conference Name: 120th Metalcasting Congress
- d. Conference Date: April 2016
- e. Conference Location: Minneapolis, MN
- f. Publication Status: Published
- g. Publication Date: April 2016
- h. Publication Identifier Type: N/A

- i. Publication Identifier: N/A
- Acknowledgement of Federal Support? Yes
- a. Title: Effect of Austenite Fraction on Rare Earth Grain Refinement
- b. Authors: Robert Tuttle
- c. Conference Name: 121st Metalcasting Congress
- d. Conference Date: April 2017
- e. Conference Location: Milwaukee, WI
- f. Publication Status: Published
- g. Publication Date: April 2017
- h. Publication Identifier Type: N/A
- i. Publication Identifier: N/A
- j. Acknowledgement of Federal Support? Yes
- Title: Effect of Rare Earth Additions on 4130
- b. Authors: Robert Tuttle
- c. Conference Name: AISTech
- d. Conference Date: May 2017
- e. Conference Location: Nashville, TN
- f. Publication Status: Published
- g. Publication Date: May 2017
- h. Publication Identifier Type: N/A
- Publication Identifier: N/A
- j. Acknowledgement of Federal Support? Yes

Book

- a. Nothing to report
- Book Chapter
 - a. Nothing to repor
- Thesis
 - Title: Characterization of Niobium Inclusions in Plain Carbon Steels
 - b. Institution: Saginaw Valley State University
 - c. Authors: Aravinda Bommareddy
 - d. Completion Date: April, 2015
 - e. Acknowledgement of Federal Support? Yes
 - Title: The Morphology of Rare Earth Inclusions in 4130 and 8630 Steels
 - b. Institution: Saginaw Valley State University
 - c. Authors: Sridhar Reddy Kottala
 - d. Completion Date: April 2017

e. Acknowledgement of Federal Support? Yes

6.

- 7. Website
 - a. Nothing to report
- 8. Other Products: Identify any other significant products that were developed under this project. Describe the product and how it is being shared.
 - a. Nothing to report